PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

Improvements in or relating to Polyamides

We, BRITISH NYLON SPINNERS LIMITED, of Pontypool, Monmouthshire Great Britain, a British Company do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the incorporation of chalk as a filler in synthetic polyamide compositions which are subsequently melt spun into textile filaments.

It is known that finely divided inorganic materials, such as silica, titanium dioxide and calcium sulphate, may be added to synthetic polyamide compositions primarily in order to produce a delustred polymer. Such materials are normally employed in amounts not in excess of about 5% by weight of the polyamide.

In addition to the foregoing inorganic materials small amounts of chalk may also be added to polyamides either as an inert extender with pigments, for example titanium dioxide, in order to obtain a stable dispersion of the pigment which is relatively free of large aggregate particles of pigment, or as a catalyst in the anionic polymerisation of caprolactam.

It has not, however, previously been proposed to incorporate large amounts of chalk, i.e. in the order of 20% by weight, as a filler in polyamides and subsequently to melt spin the polymer to give acceptable textile filaments.

One reason for this may be that the presence of substantial amounts of chalk in some polyamides, particularly those containing adipic or substituted adipic acid residues, results in considerable difficulties in melt spinning owing to the formation of excessive amountse of gel and other undesirable degradation products in the melt pool prior to extrusion when the residence time of a portion of the polymer in the melt pool is long. Such products are frequently the cause of blockages in the filter pack and also result in the production of a yarn which suffers excessive filamentation at drawing and which is of a generally unacceptable quality.

It is thought that the formation of gel may be caused by a small amount of calcium adipate being formed during blending of the molten polymer and chalk and that this compound cyclises whilst held at melt spinning temperatures in the melt pool to produce cyclopentanone, which compound is considered to be at least partially responsible for the formation of the gel and other degradation products in the melt pool.

Another reason is that the addition of substantial amounts of chalk to polyamides during polymerisation markedly decreases the rate of polymerisation and may even completely inhibit it if added in the initial stages.

We have now discovered that considerable amounts of chalk can be sufficiently well dispersed in polyamides to yield polymers that can be spun into filaments capable of being drawn to a draw ratio as high as 4.6 on conventional equipment to provide acceptable textile fibres, provided that the chalk is added to the molten polyamide not earlier than the end of the polymerisation process, and that, particularly where the polyamide contains adipic and substituted adipic acid residues, for example polyhexamethylene adipamide, the time during which the chalk containing polyamide is in the molten state is minimal.

According to one aspect of the present invention there are provided filaments of linear synthetic high molecular weight polyamides containing at least 10% by weight of chalk uniformly dispersed therein, the chalk having an average particle size not greater than 5 microns.

Preferably the filaments should contain 20%—35% by weight of chalk and preferably also the average particle size of the chalk should

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be not greater than 0.1 microns in order that the spinning and drawing performance shall not be impaired by the presence of agglomerates of particles of larger particle size.

According to another aspect of this invention there is provided a process for the manufacture of the aforesaid filaments wherein the chalk is uniformly dispersed in the molten polyamide after the final stage in the polymerisation cycle and the polyamide containing the chalk subsequently formed into filaments.

The chalk containing polyamides are preferably spun into filaments by the conventional melt spinning process, although it is envisaged that other forms of spinning such as plasticised melt spinning or solution spinning might be employed provided that, in the instance of solution spinning for example, the added matter (solvent) does not react with the chalk.

The filaments of this invention and the process for their manufacture are illustrated in the following example in which the parts are parts by weight. It is to be understood that this example is not intended in any way to limit the scope of the invention.

Example

A. Preparation of polymer chalk mixture

A four litre stirred autoclave is charged with ξ-caprolactam, 1500 parts, and water, 500 parts. The vessel is heated to 220°C. over a period 2 1/3 hours, during which time the pressure in the autoclave rises to 250 p.s.i., and then to 260°C. over a period of 1 hour the pressure being reduced to atmospheric during this period. The melt is then heated for a further period of 1½ hours at a temperature of 260—290°C. at atmospheric pressure and under an atmosphere of nitrogen. Precipitated calcium carbonate, 300 parts (20% by weight) of a grade sold under the trade mark of Calofort U (average particle size 0.1μ) by John and E. Sturge Limited, Birmingham, freed from entrained oxygen by purging with nitrogen, is then added to the stirred melt and the mixture stirred for 1¼ hours under at atmosphere of nitrogen to effect uniform dispersion. The polymer-chalk mixture is extruded, chipped and washed with boiling water for 30 hours to remove ξ-caprolactam and other water soluble material. The polymer chips are then dried at 60°C. and 100 millimetres mercury pressure for 16 hours.

B. Melt spinning polymer-chalk mixture

The polymer-chalk mixture is melt spun at 265°C. through a twenty hole spinneret on conventional melt spinning apparatus. The filaments are cooled by a transverse blast of air in the usual manner and wound up at 1012 feet per minute. The as spun yarn is cold drawn at a draw ratio of 4.6 to yield a yarn having a tenacity of 3.7 gm. per denier and 10% extensibility. At a draw ratio of 3.7 the yarn has a tenacity of 2.1 gm. per denier and 30% extensibility.

Other additives such as antioxidants or pigments etc. may be added during or after polymerisation in the usual manner.

Other polymers which are particularly suitable for carrying out the process of this invention include polyhexamethylene sebacamide, polyaminoheptanoic acid and polyamino-undecanoic acid.

Since all the particles of chalk contained in the yarn are covered by polymer and do not project through the surface of the yarn, the properties of the yarn are substantially unimpaired; thus it has the abrasion resistance and resistance to solvents normally associated with wholly polyamide yarns. Thus for example, polyamide filaments containing 20% by weight of chalk may be contacted with dilute hydrochloride or acetic acids without any significant loss in tenacity being observed compared with similarly treated filaments which do not contain the chalk.

The incorporation of substantial amounts of chalk in polyamide yarns makes possible the production of a cheaper yarn which still has acceptable textile properties. This cost reduction is particularly important in end uses where polymer cost is the major item in the cost of the product, e.g. in staple fibre end uses.

WHAT WE CLAIM IS:—

1. Filaments of linear synthetic high molecular weight polyamides containing at least 10% by weight of chalk uniformly dispersed therein, the chalk having an average particle size not greater than 5 microns.

2. Filaments according to Claim 1 containing 20—35% by weight of chalk.

3. Filaments according to Claims 1 or 2 wherein the average particle size of the chalk contained therein is not greater than 0.1 microns.

4. Filaments according to Claim 1, 2 or 3 wherein the said filaments have been drawn to at least three times their original length.

5. Staple fibres produced from the filaments of any one of the preceding claims.

6. Filaments or staple fibres according to any one of the preceding claims wherein the polyamide is polyhexamethylene adipamide.

7. Filaments or staple fibres according to any one of claims 1—5 wherein the polyamide is polyepsilon caprolactam.

8. A process for the manufacture of the filaments of Claim 1 wherein the chalk is uniformly dispersed in the molten polyamide after the final stage in the polymerisation cycle and the polyamide containing the chalk subsequently formed into filaments.

9. A process for the manufacture of the fialments of Claim 1 wherein the chalk is uniformly dispersed in the molten polyamide polymerisation at the end of the final stage in the polymerisation cycle and the polyamide

containing the chalk is subsequently formed into filaments.

10. A process according to Claims 8 or 9 wherein 20—35% by weight of chalk is uniformly dispersed in the molten polyamide.

11. A process according to any one of Claims 8, 9 or 10 wherein the average particle size of the chalk is not greater than 0.1 microns.

12. A process according to any one of the preceding claims 8—11 wherein the chalk containing polymer is formed into filaments by the method of melt spinning and the filaments are solidified by a transverse blast of air and subsequently wound up onto a package.

13. A process according to any one of Claims 8—12 wherein the as spun filaments are drawn to at least three times their original length.

14. A process according to any one of the preceding Claims 8 to 13 wherein the polyamide is polyhexamethylene adipamide.

15. A process according to any one of Claims 8 to 13 wherein the polyamide is polyepsilon caprolactam.

16. A process for the manufacture of the filaments of Claim 1 substantially as described herein with reference to the Example.

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